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Temperature, Moisture, and Strain Rate Effects on the Compressive Mechanical Behavior of Nylon 6/6

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Temperature, Moisture, and Strain Rate Effects on the Compressive Mechanical Behavior of Nylon 6/6

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ABSTRACT

Material test results are presented for the mechanical behavior of Nylon 6/6 in compression. Static compression modes include direct compression, stress relaxation and creep. Dynamic direct compression results are included. Tests are performed at atmospheric pressure; strain rates range from 10^{-4}/sec to 10^{2}/sec; temperatures are 20, 65, 110, 150 and 200 deg C; moisture levels are 0% (dry), 2% and 6% (saturated); true strains to -0.25 are imposed. Our empirical Temperature-Moisture equivalence of 14 deg C per 1% moisture is discussed with respect to the "free volume" concept.

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TEMPERATURE, MOISTURE AND STRAIN RATE EFFECTS ON THE COMPRESSIVE MECHANICAL BEHAVIOR OF NYLON 6/6

INTRODUCTION AND SCOPE

Materials testing is essential to the development and verification of constitutive models for material deformation. Our purpose is to generate a consistent set of data from which nonlinear visco-elastic constitutive models of Nylon 6/6 can be constructed and evaluated. We use 3-dimensional plotting liberally to provide the reader a quick feel for the data; researchers are welcome to contact us for copies of datafiles for more rigorous applications. Research on the effects of hydrostatic pressure is in progress and will be documented separately.

Individual citations on the mechanical behavior of Nylon 6/6 are typically restricted in their temperature range, strain or strain-rate regime, or testing mode. Variability in material stock and testing techniques further frustrate efforts to patch together a consistent comprehensive set of data for material modeling purposes. We present data from a single batch of material stock at five temperatures (20, 65, 110, 150, 200 deg C), seven strain rates (10^{-4} to 10^{2}/sec), three moisture levels (0%, 2% and 6%) and three deformation modes ~~(direct com~~pression, compressive stress relaxation and compressive creep).

Finally, we quantify the comparable effects of temperature and moisture on the compressive behavior of Nylon 6/6, concluding that a reasonable equivalence in direct compression is 14 deg C per 1% moisture content. This value also carries over reasonably well in compressive stress relaxation and creep.

MATERIAL AND SPECIMEN PREPARATION

MATERIAL: Specimens are extracted from 5" O.D. x 2.75" I.D. x 13" long tube stock of DuPont Zytel 101 material extruded and annealed by Polymer Corporation; material specifications are in the Appendix.

GEOMETRY: Specimens are cylindrical, nominally 0.500" diameter by 0.750" tall; the two parallel faces are concentrically grooved (Appendix) to trap lubricant (petroleum jelly at room temperature and Teflon powder otherwise) and minimize compressive barreling. Specimen axes are parallel to the stock axis.

MOISTURE: Dry (0% moisture) specimens are prepared by baking them out at 100 deg C for two weeks, followed by storage in dessicated jars. The 2% moisture (equivalent to 50% RH) samples are conditioned by boiling as-machined specimens in potassium acetate solution for a week within a reflux condenser as per the technique recommended by DuPont [1]. The 6% moisture (fully saturated) specimens are conditioned by boiling in distilled water for two weeks. Moisture levels are determined by weighing. The 2% and 6% content specimens are stored at room temperature in their respective solutions.

STATIC AND DYNAMIC TESTING TECHNIQUES

Test approach, hardware and software details are documented elsewhere [2]. Direct compression up to rates of 10^{-1}/sec is performed at constant true strain rate; tests at higher rates were more closely performed at constant engineering strain rate due to equipment limitations. The loading portion of the stress relaxation tests are done at a strain rate of 10^{-2} /sec. The creep tests are nominally loaded at a rate of 2 ksi per second; thereafter, load is continually increased per current deformation to maintain constant true stress (1 ksi = 1,000 psi = 6.895 MPa).

RESULTS FOR DIRECT COMPRESSION

STRESS VS. STRAIN AND STRAIN RATE

The results for direct compression of Nylon 6/6 are grouped into 3 sets of plots corresponding to 0%, 2% and 6% moisture content respectively. Each set consists of 5 plots for 20, 65, 110, 150 and 200 deg C. (We henceforth delete the obvious negative signs for stress and strain). Refer to Figures 1,2,3. Collectively, these data suggest the following conclusions:

a. For a fixed moisture content, temperature lowers the overall strength, elastic modulus and "strain-rate sensitivity" of Nylon 6/6. This last feature allows us to eliminate several elements of the test matrix, especially at the higher temperatures.

b. Analagously, at a fixed temperature, moisture lowers the overall strength and modulus of Nylon 6/6. The similar effects of temperature and moisture are investigated in a later section.

We briefly elaborate on the conclusions "a." above:

The lowering of overall strength and elastic modulus by temperature is clearer if we arbitrarily focus on a fixed strain rate (say, 10^{-1}/sec) and moisture level (say, 0% moisture). Under these conditions, the effect of temperature is apparent from Figure 4a. Similarly, Figures 4b,c illustrate the effect of temperature at moisture levels of 2% and 6% respectively. These 3 figures show that the effect of temperature on strength and modulus is more profound for Nylon 6/6 having low moisture content. A plot of the elastic modulus vs. temperature and strain rate is presented in the next section.

By "strain-rate sensitivity" we mean the variation in stress with respect to strain rate at fixed temperature, moisture, and strain. Graphically, we have replotted the data of Figures 1a, 2a, and 3a (20 deg C, 0%, 2%, 6% moisture) in Figures 5a,b,c respectively. Each curve traces how the value of stress at a selected value of strain (0.05, 0.10, 0.15, 0.20, 0.25) increases with strain rate. In conclusion "a." above, we mean that similar sets of curves at elevated temperatures would appear more flat or horizontal.

ELASTIC MODULUS VS. TEMPERATURE AND STRAIN RATE

Strictly speaking, viscoelastic materials like Nylon 6/6 have no classical linear elastic regime; nevertheless we operationally define an Elastic Modulus from the direct compression data to characterize the effects of Temperature and Moisture on material stiffness. Our Elastic Modulus definition is the slope of true stress vs. true strain, such that the upper bound of true stress does not exceed 20% of the maximum true stress for the entire test. The dynamic test configuration for strain rates beyond 10^{-1} sec cannot provide sufficiently accurate small strain data for modulus determination.

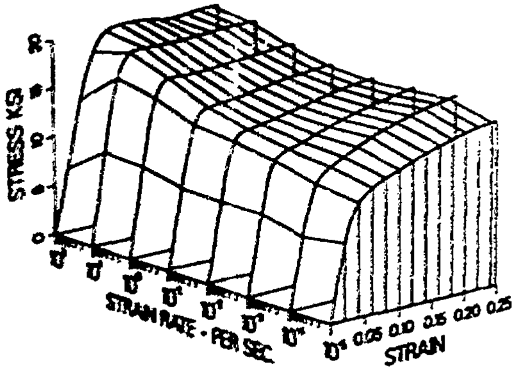
Elastic Modulus as a function of Temperature and Strain Rate is plotted in Figure 6 for the 0% Moisture (upper surface) and 2% Moisture (lower surface) conditions (note: 1 Msi = 10^6psi). While temperature and moisture both lower the Elastic Modulus as anticipated, the influence of moisture is less pronounced at higher temperatures; this concurs with the trend of the flexural modulus for Zytel 101 nylon resin reported by DuPont [1]. Although our test matrix does not include enough tests on fully saturated material at elevated temperatures to warrant plotting their moduli in this fashion, Figure 4c indicates that the overall flattening effect of moisture continues.

FIGURE 1:

STRESS VS. STRAIN
AND STRAIN RATE
AT VARIOUS TEMPERATURES

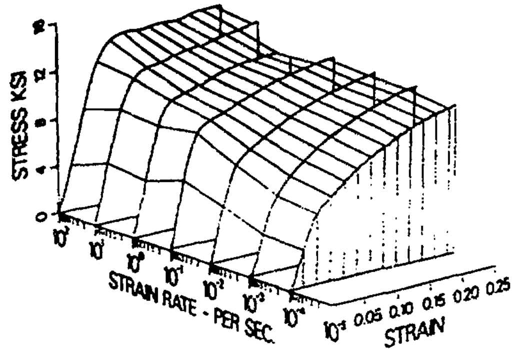
(DRY NYLON 6/6)

NYLON 6/6, 20 DEG. C., 0% MOISTURE, COMPRESSION



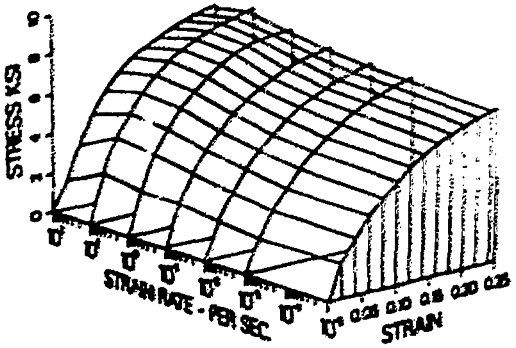
(1a)

NYLON 6/6, 65 DEG. C., 0% MOISTURE, COMPRESSION



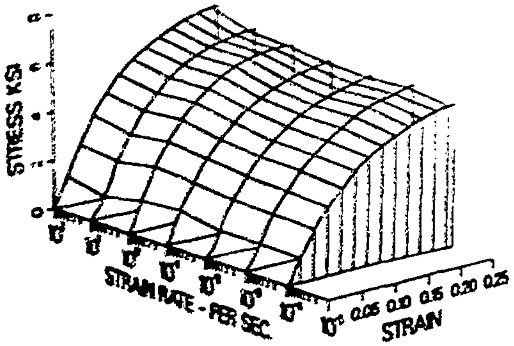
(1b)

NYLON 6/6, 110 DEG. C., 0% MOISTURE, COMPRESSION



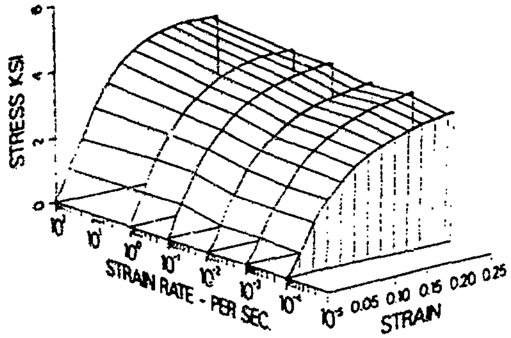
(1c)

NYLON 6/6, 150 DEG. C., 0% MOISTURE, COMPRESSION



(1d)

NYLON 6/6, 200 DEG. C., 0% MOISTURE, COMPRESSION



(1e)

FIGURE 2:

STRESS VS. STRAIN
AND STRAIN RATE
AT VARIOUS TEMPERATURES

(2% MOISTURE)

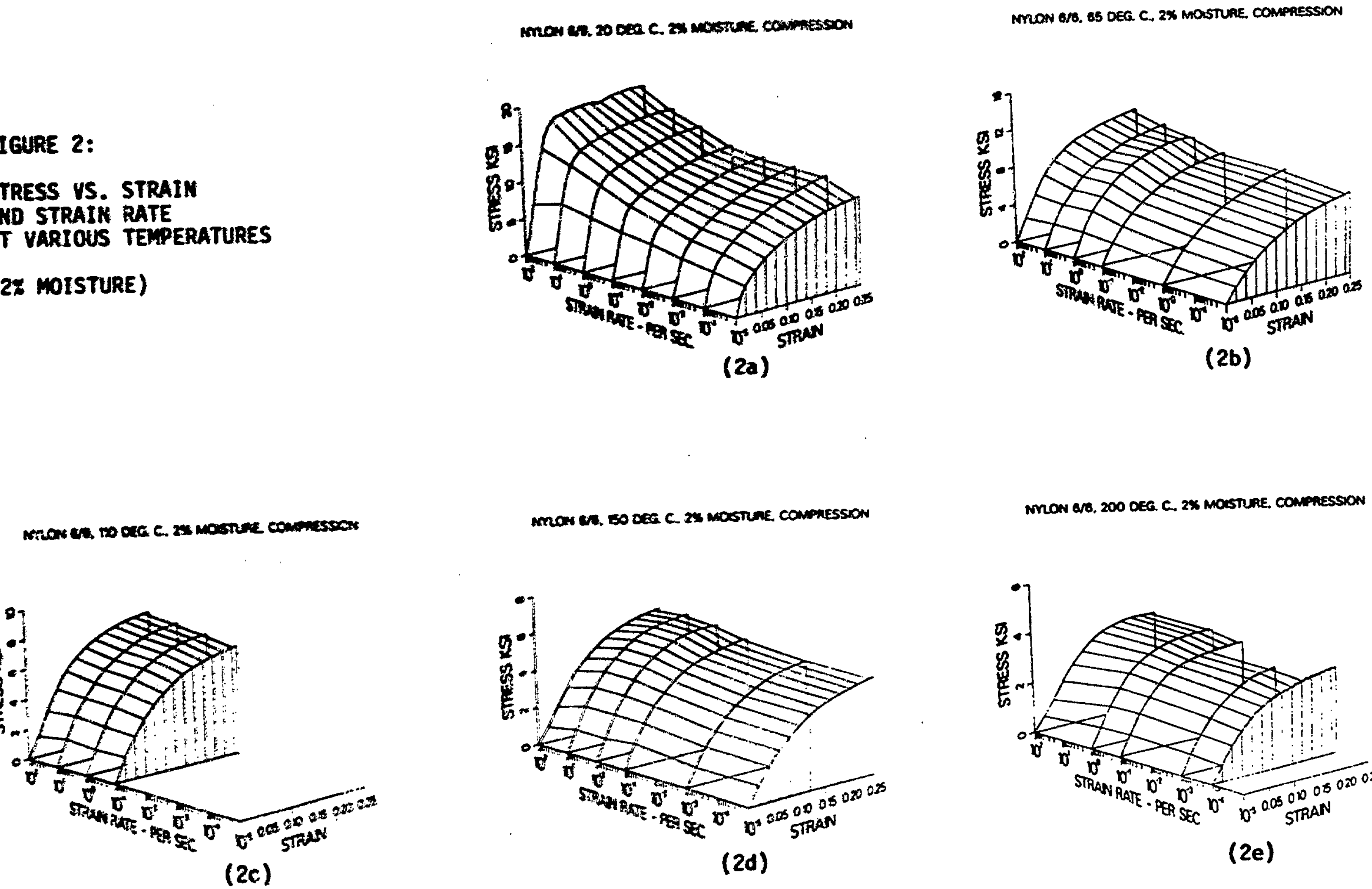
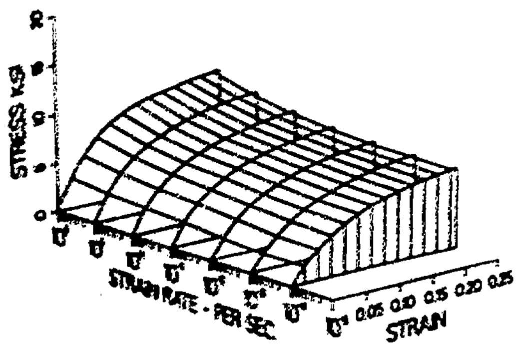


FIGURE 3:

STRESS VS. STRAIN
AND STRAIN RATE
AT VARIOUS TEMPERATURES

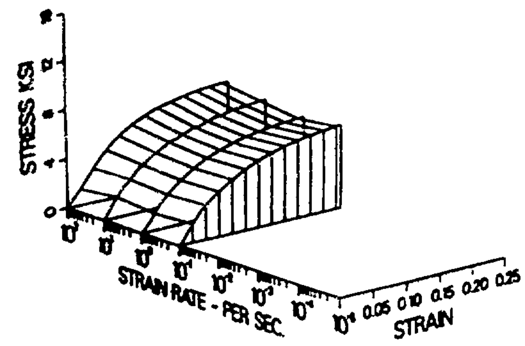
(6% MOISTURE)

NYLON 6/6, 20 DEG. C., 6% MOISTURE, COMPRESSION



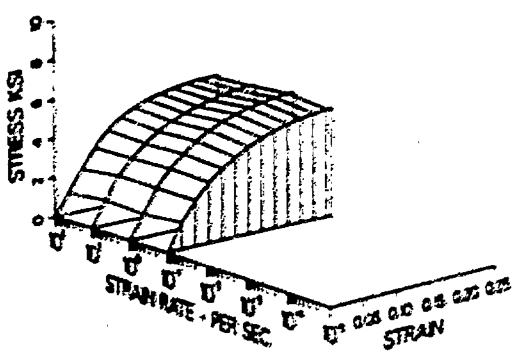
(3a)

NYLON 6/6, 65 DEG. C., 6% MOISTURE, COMPRESSION



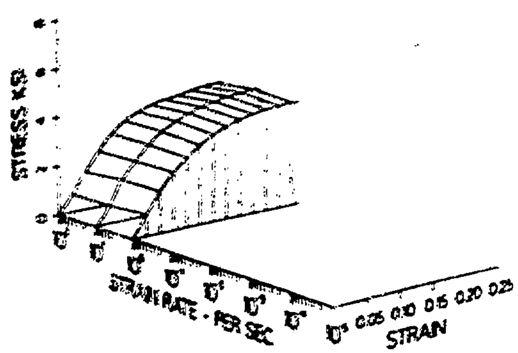
(3b)

NYLON 6/6, 110 DEG. C., 6% MOISTURE, COMPRESSION



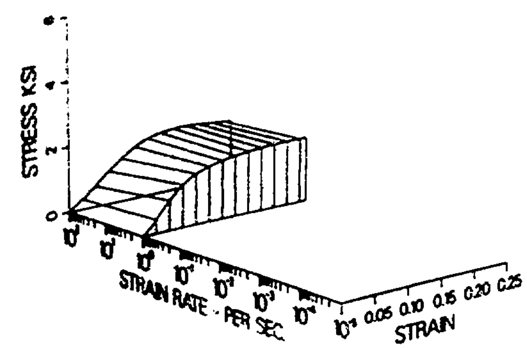
(3c)

NYLON 6/6, 150 DEG. C., 6% MOISTURE, COMPRESSION



(3d)

NYLON 6/6, 200 DEG. C., 6% MOISTURE, COMPRESSION



(3e)

FIGURE 4:

EFFECTS OF TEMPERATURE AND MOISTURE AT FIXED STRAIN RATE $(10^{-1}/\text{sec})$

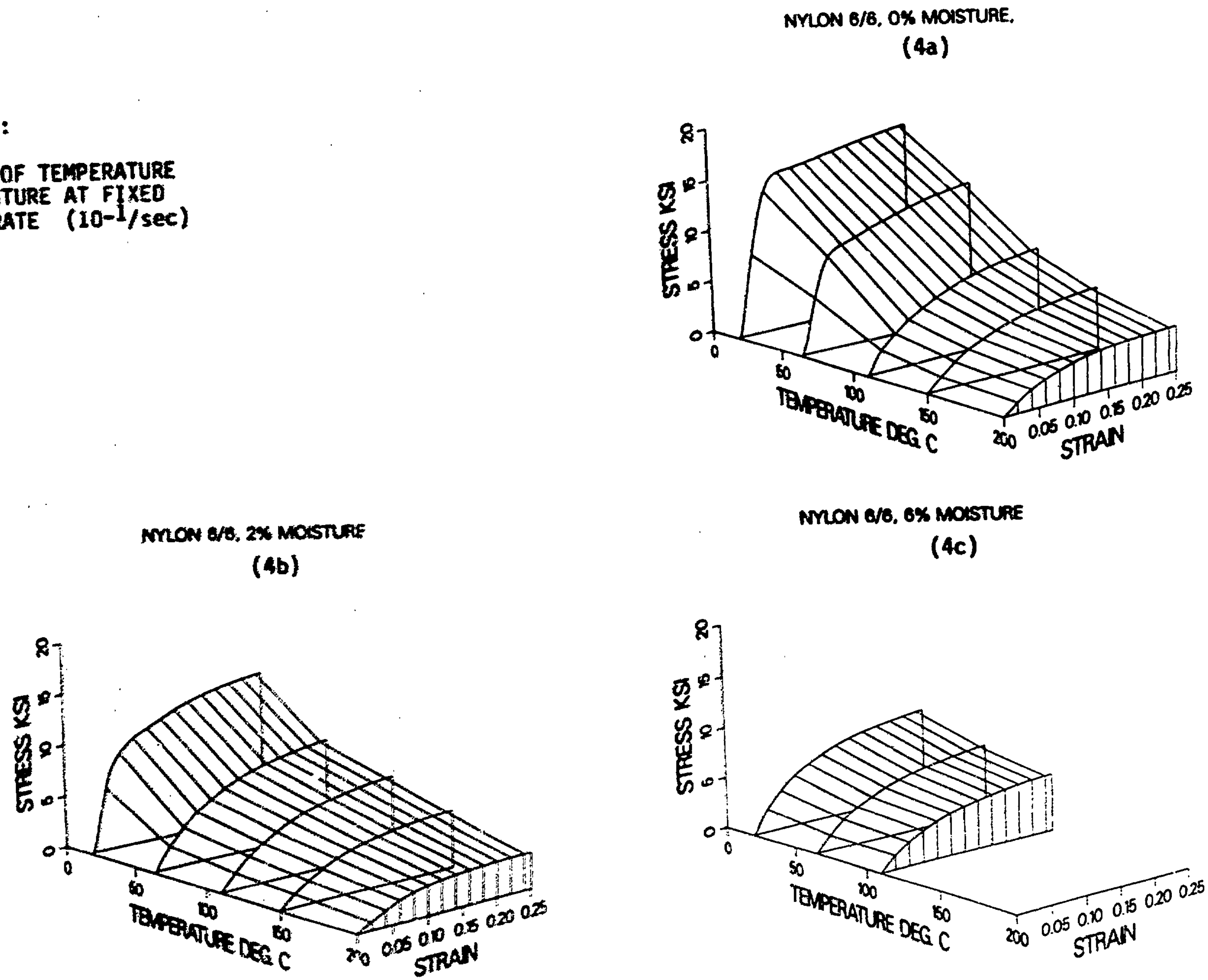
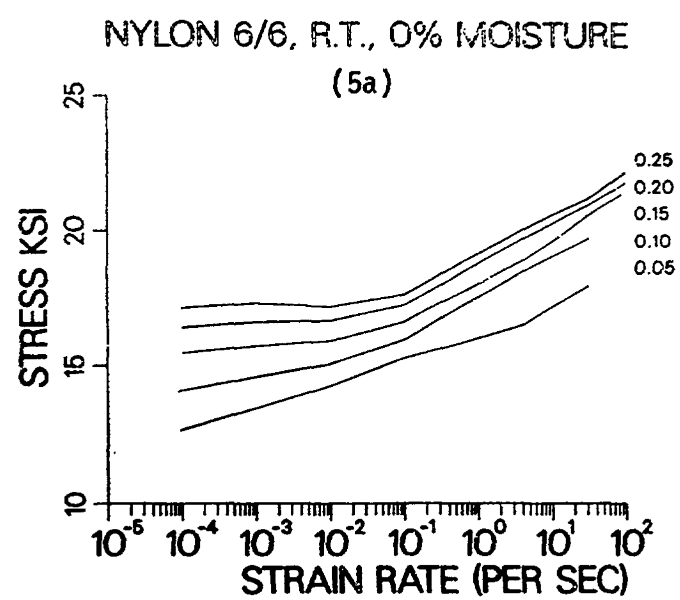
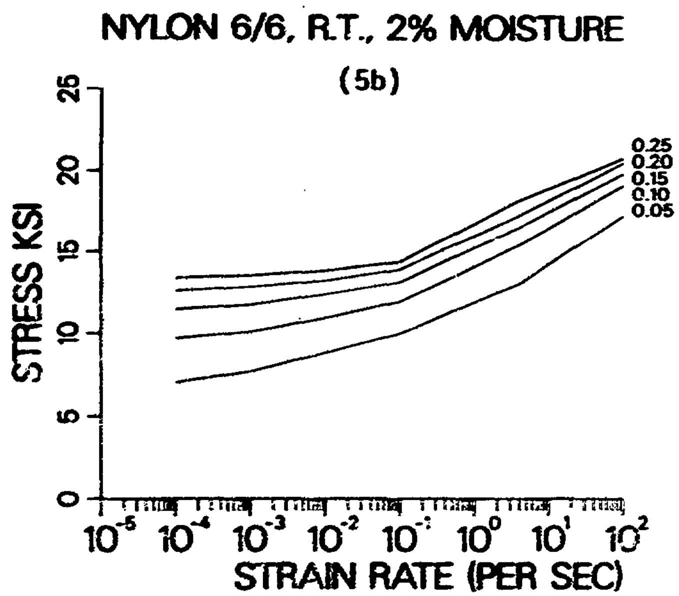


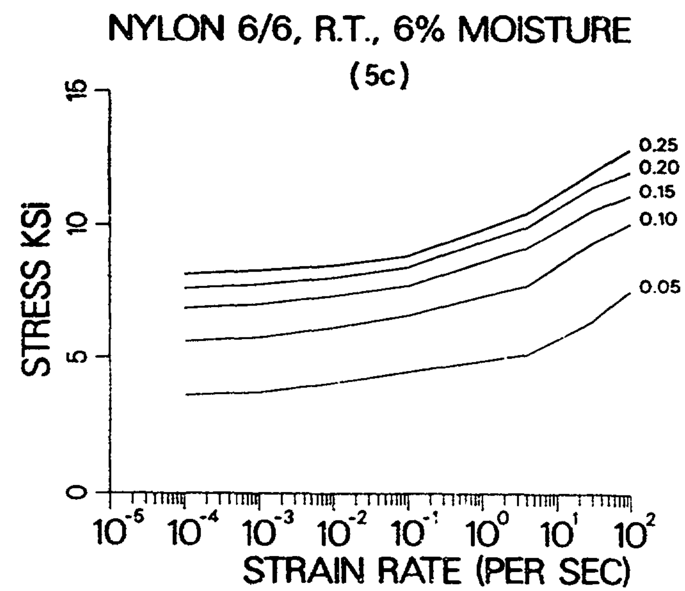
FIGURE 5:

ILLUSTRATION OF
STRAIN-RATE SENSITIVITY

(20 Deg. C)







NYLON 6/6 COMPRESSIVE MODULUS, 0% & 2% MOISTURE

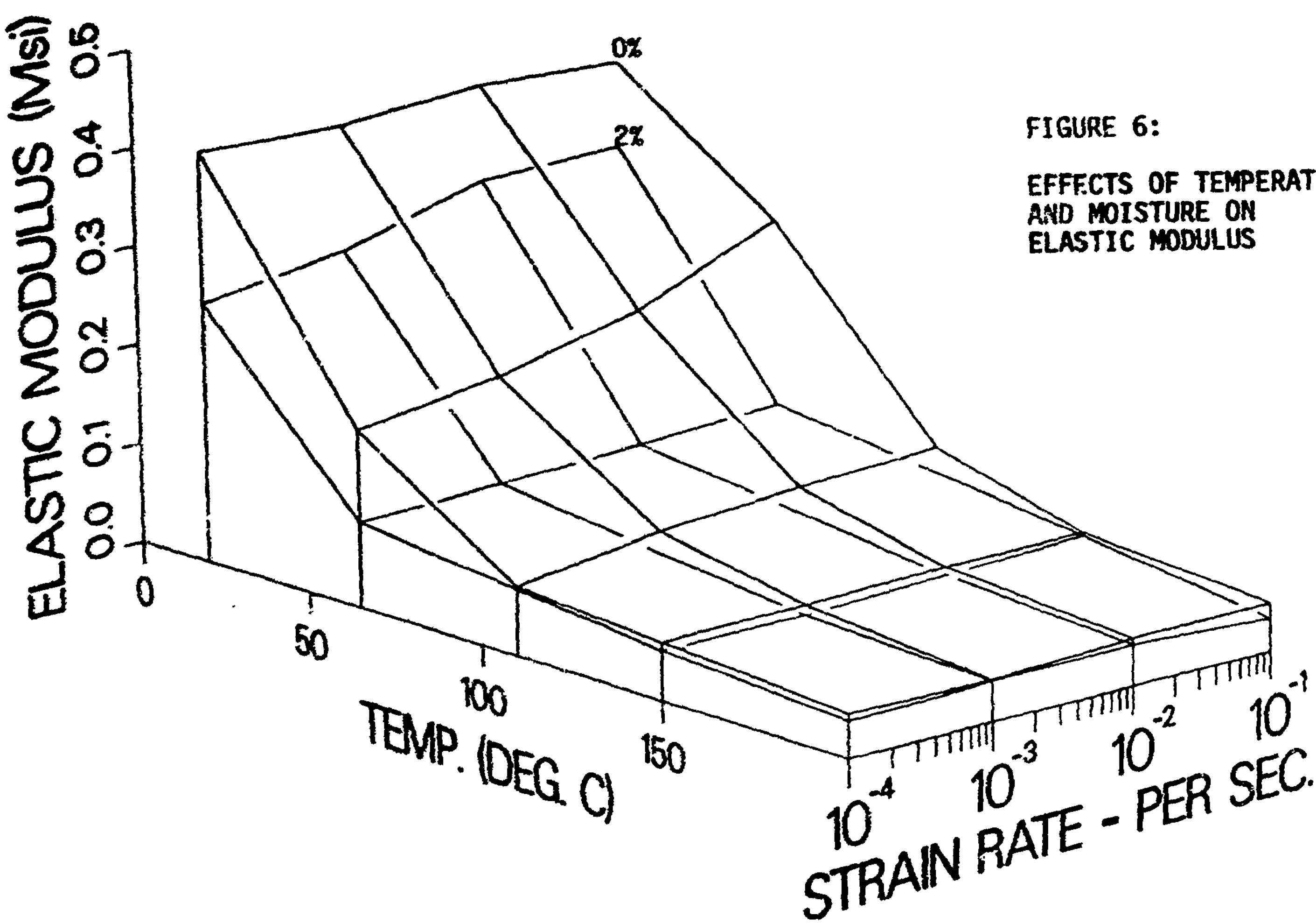


FIGURE 6:

EFFECTS OF TEMPERATURE AND MOISTURE ON ELASTIC MODULUS

RESULTS FOR STRESS RELAXATION

TYPICAL STRESS VS. TIME BEHAVIOR

Compressive stress relaxation tests are performed on dry Nylon 6/6 at 20, 65, 110, 150 and 200 deg C to fixed strains of 0.025 (linear) and 0.20 (nonlinear). A smaller test matrix was performed on 2% moisture Nylon 6/6 at the same temperatures and typically to a strain of 0.15 (nonlinear). The nominal strain rate during loading was 10^{-2}/sec; test durations were 500 or 1000 seconds. Supplementary tests on 6% moisture Nylon 6/6 were performed at 20 deg C to investigate the equivalencing of temperature and moisture; these are discussed in a later section.

A typical stress relaxation plot is shown in Figure 7a for dry Nylon 6/6 at 20 deg C compressed to strains of 0.025, 0.10 and 0.20 respectively. The "knee" in the latter (upper) two curves is due to yielding of the material; i.e. up to the peak stresses the time axis is proportional to strain since loading is at constant strain rate. Similarly, Figure 7b illustrates stress relaxation for 2% moisture Nylon 6/6 at 20 deg C compressed to strains of 0.05, 0.15 and 0.20; Figure 7c shows stress relaxation for 6% moisture Nylon 6/6 at 20 deg C to strains of 0.025, 0.10 and 0.20 respectively.

RELAXATION MODULUS VS. TIME

For viscoelastic materials, the instantaneous stress state is influenced by its entire deformation history. Constitutive models of this hereditary material behavior are often characterized by superposition integrals whose integrands involve the material relaxation modulus $E(t)$, i.e. stress to strain ratio as a function of time. Conceptually then, a realistic material model would be able to predict our constant strain-rate direct compression results from knowledge of the relaxation modulus.

We have recouched the stress vs. time data of the preceeding section in terms of the relaxation modulus, $E(t)$. Figures 8a,b are the relaxation moduli for dry Nylon 6/6 at a total strain of 0.025 and 0.20 respectively. Corresponding relaxation moduli for Figure 8a lie above those of Figure 8b since the latter are performed deep within the region of nonlinear material behavior. (The dashed lines are discussed later.) Figure 8c is the relaxation modulus plot for 2% moisture Nylon 6/6 at 0.15 total strain.

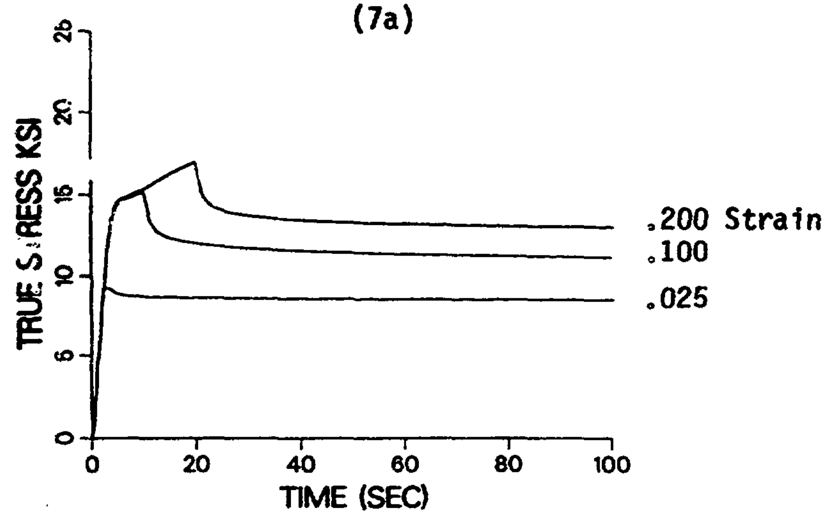
FIGURE 7:

TYPICAL STRESS VS. TIME
BEHAVIOR IN STRESS
RELAXATION TESTS

(20 Deg. C)

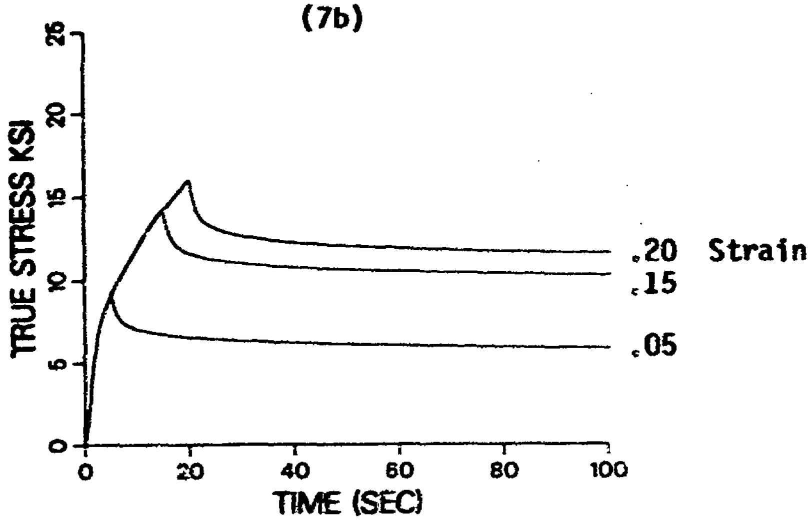
STRESS RELAXATION, NYLON 6/6, R.T., 0% MOISTURE

(7a)



STRESS RELAXATION, NYLON 6/6, R.T., 2% MOISTURE

(7b)



STRESS RELAXATION, NYLON 6/6, R.T., 6% MOISTURE

(7c)

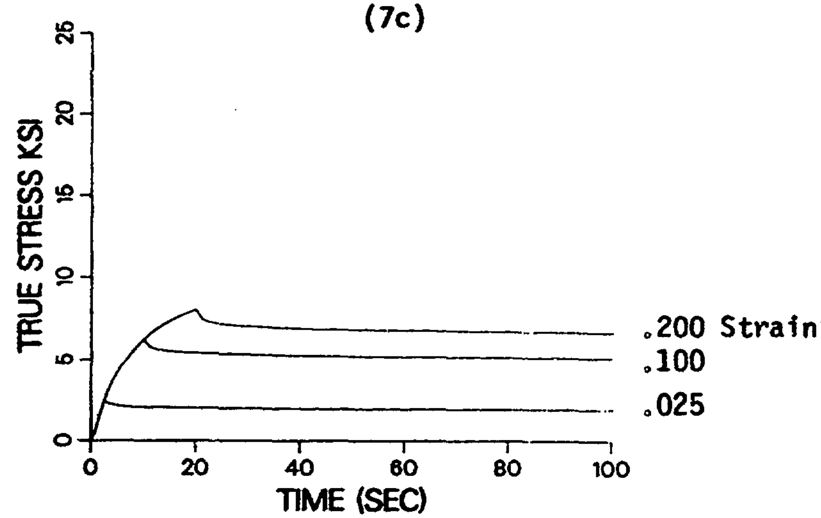
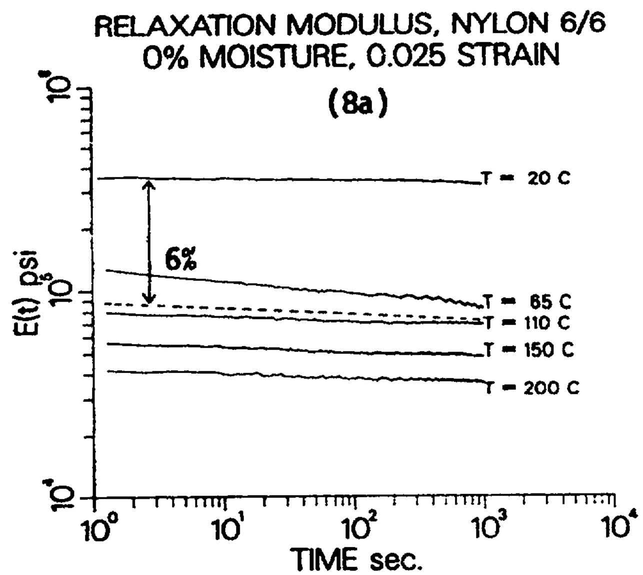
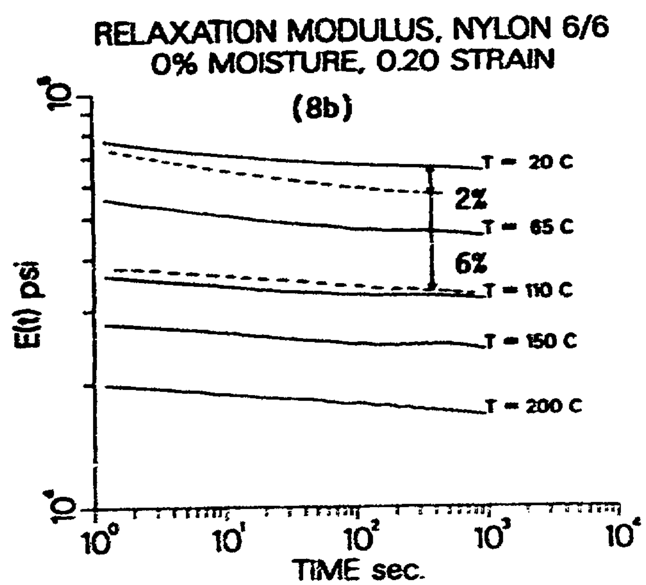
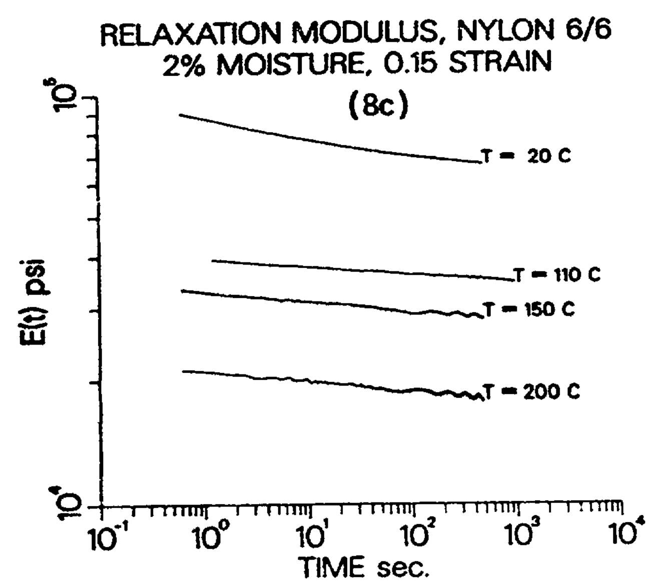


FIGURE 8:

RELAXATION MODULUS VS. TIME AT VARIOUS CONDITIONS







RESULTS FOR CREEP TESTING

TYPICAL STRAIN VS. TIME BEHAVIOR

Creep is the time-dependent deformation of a material under stress. Our compressive creep tests were typically loaded at a rate of 2 ksi/sec and held for 5 hours. During the test, the load is continually increased per the current deformation to impose a constant true stress, using a Poisson Ratio of 0.41 for all tests. Because non-zero moisture contents cannot be guaranteed to remain constant at elevated temperatures for tests of long duration, most creep tests were performed on 0% (dry) condition Nylon 6/6. Supplementary creep tests were performed on 2% and 6% moisture Nylon 6/6 to investigate the equivalencing of temperature and moisture; these are discussed in a later section.

A typical creep strain vs. time plot is shown in Figure 9a for dry Nylon at 20 deg C and stress levels of 6 and 12 ksi. The data indicates that increasing stress causes increases in initial strain, primary creep duration and secondary creep rate. Similar conclusions hold for 2% moisture Nylon 6/6, Figure 9b (3, 6 and 12 ksi) and also for 6% moisture Nylon 6/6, Figure 9c (2 and 6 ksi).

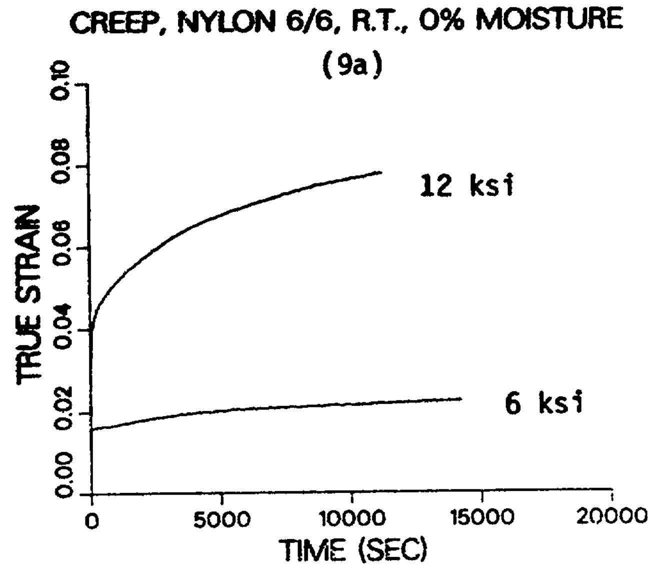
CREEP COMPLIANCE VS. TIME

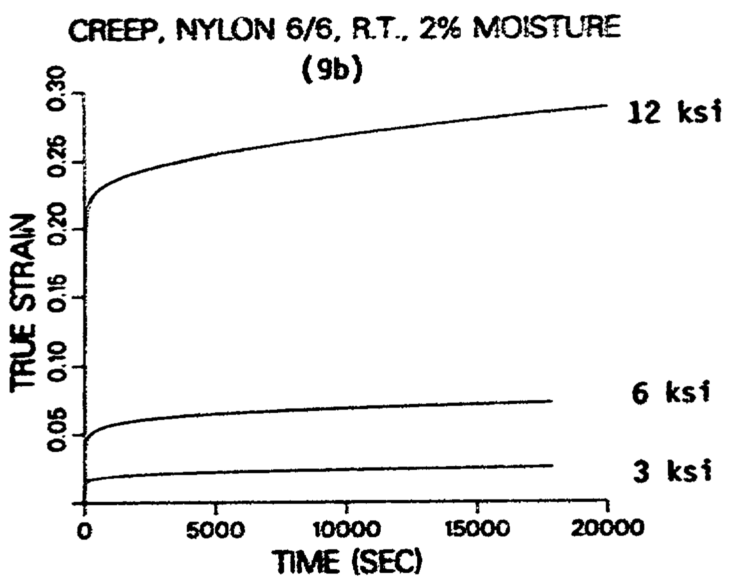
Analagous to our discussion for the relaxation modulus, the deformation state of a viscoelastic material is influenced by its entire stress history. The corresponding hereditary integral describing this behavior involves the material creep compliance $D(t)$ or $J(t)$, the strain to stress ratio as a function of time.

Our creep strain vs. time data has been replotted in terms of creep compliance vs. time for dry Nylon 6/6 at stresses of 4 ksi and 6 ksi in Figures 10a,b respectively. (The dashed lines are discussed later.)

FIGURE 9:

TYPICAL STRAIN VS. TIME BEHAVIOR IN CREEP TESTS (20 Deg. C)





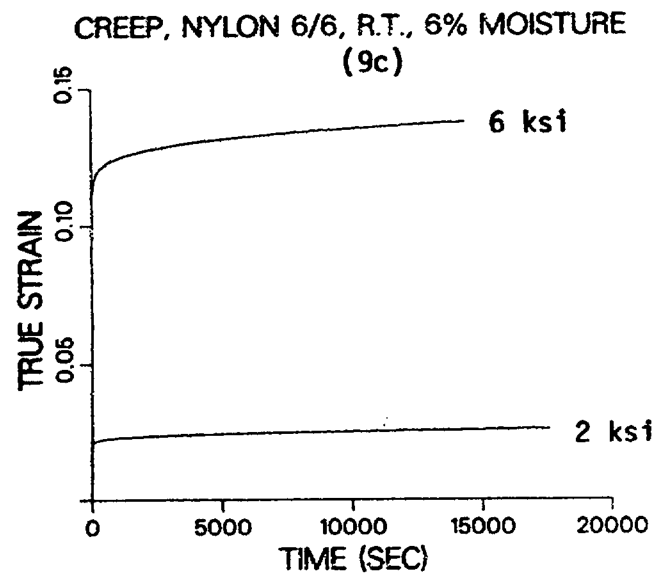
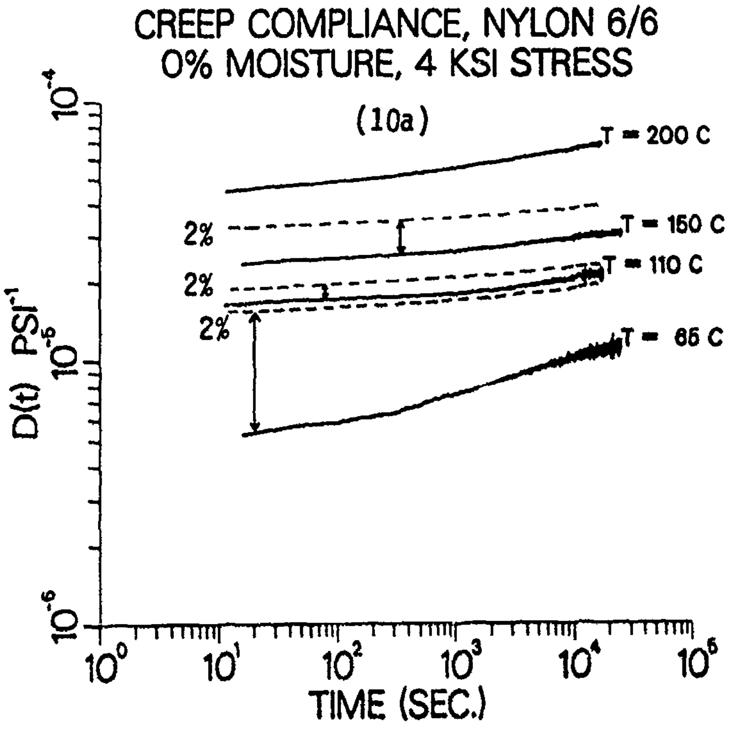
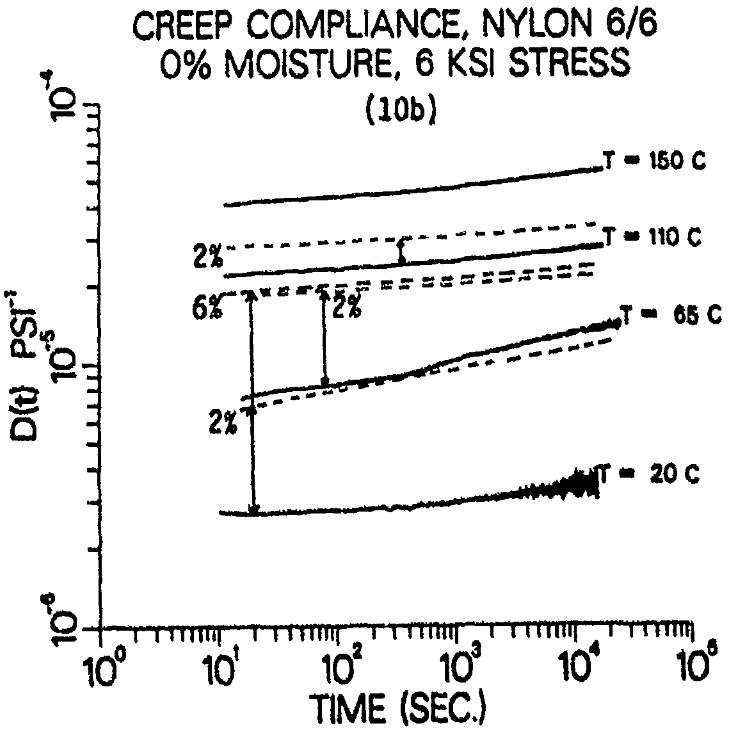


FIGURE 10:

CRFEP COMPLIANCE VS. TIME
AT VARIOUS CONDITIONS





TEMPERATURE-MOISTURE EQUIVALENCING

EMPIRICAL RESULTS IN THE THREE COMPRESSION MODES

The motivation for establishing an equivalencing relationship between the influences of temperature and moisture on the mechanical behavior of Nylon 6/6 is twofold. From a materials science aspect, such a relationship can assist in understanding the fundamental microscopic mechanisms of polymer deformation; for example, as discussed in the next section. Second, from an engineering viewpoint, the mechanical behavior of a polymer at some temperature and moisture level can be predicted from test data already documented on dry polymer at some equivalent (higher) temperature.

Our tests cover three different deformation modes (direct compression, stress relaxation and creep). Guided by the intended application for our tests, we decide to obtain the best-fit empirical temperature-moisture relationship for the direct compression mode then check to see whether behavior in the stress relaxation and creep modes are consistent with this relationship.

DIRECT COMPRESSION: Our empirical fit of 100 deg C per 7% moisture (14 deg C per 1% moisture) is illustrated in Figure 11a. The solid stress-strain curves correspond to 0% (dry) Nylon 6/6 direct compression data at temperatures from 20 deg C on up. The two dashed stress-strain curves are data for Nylon 6/6 at 20 deg C but at moisture contents of 2% and 6%, plotted along the temperature axis using the 100 deg C per 7% moisture equivalencing factor (e.g. the 2% curve is placed at (100/7)x2 + 20 = 48.6 deg C). The important point is that the behavior at a given temperature and moisture content can be approximated by that of dry material at an equivalent (higher) temperature. We see that the predicted stress-strain curve based on dry data is somewhat high for the 2% condition but only very slightly low for the 6% condition.

Similarly, Figure 11b illustrates the empirical fit, where the two dashed curves are data at 65 deg C for 2% and 6% moisture levels; the error is slightly high for the 2% case and slightly low for the 6% case. Figure 11c illustrates the fit where the dashed curves are data at 110 deg C for 2% and 6% moisture; Figure 11d illustrates the fit where the dashed curve is data at 150 deg C for 2% moisture. The same value of 100 deg C per 7% moisture is used for all four figures; comparable results hold for other strain rates as well.

STRESS RELAXATION: Having established the 14 deg C/ 1% moisture equivalencing factor from direct compression data, we check if the stress relaxation behavior is consistent with this factor. Returning to the relaxation modulus plot in Figure 8a, the dashed line corresponds to data at 20 deg C and 6% moisture. This would be an equivalent temperature for dry data of (100/7)x6 + 20 = 106 deg C; this agrees reasonably with its relative location in the figure. In a similar manner but for strains in the nonlinear region, the two dashed lines in Figure 8b correspond to data at 20 deg C and moistures of 2% and 6%; the former has a calculated equivalent temperature of 49 deg C so the dashed line (actual behavior) is higher in position than predicted. Overall, the equivalencing factor provides a reasonable approximation for relaxation behavior of Nylon 6/6.

CREEP: Finally, we investigate whether the creep behavior is consistent with our equivalencing factor. Returning to Figure 10a, the 3 dashed curves are, in ascending order, data for Nylon 6/6 at (65 deg C, 2% moisture), (110 deg C, 2% moisture) and (150 deg C, 2% moisture). Their calculated equivalent temperatures are 94 deg C, 139 deg C and 179 deg C. The first dashed curve is somewhat higher than predicted, the cther two somewhat lower. In Figure 10b, the 4 dashed curves in ascending order are data at (20 deg C, 2% moisture), (65 deg C, 2% moisture), (20 deg C, 6% moisture) and (110 deg C, 2% moisture). Their calculated equivalent temperatues are 49 deg C, 94 deg C, 106 deg C and 139 deg C respectively; actual behavior indicated by the dashed lines is somewhat high for the first two and low for the last two. Hence, the predictions based upon our equivalencing factor straddle both sides of the actual behavior and provide a reasonable approximation to the creep behavior of Nylon 6/6.

CONCEPT OF "FREE VOLUME"

The search for a physically-based explanation for the equivalent effects of temperature and moisture on the mechanical behavior of polymers has drawn attention to the concept of "free volume", defined as the difference between the specific volume and occupied volume per gram. Conceptually, increasing the system's free volume (whether by raising the temperature or moisture content) amplifies polymer segmental mobilities.

The experiments of Doolittle [3] describing the temperature dependence of viscosity in liquids expressed in terms of free volume has been applied to linear viscoelastic polymeric systems by Williams, Landel and Ferry [4] as:

$$ln a_{12} = B\left(\frac{1}{f_2} - \frac{1}{f_1}\right) \tag{1}$$

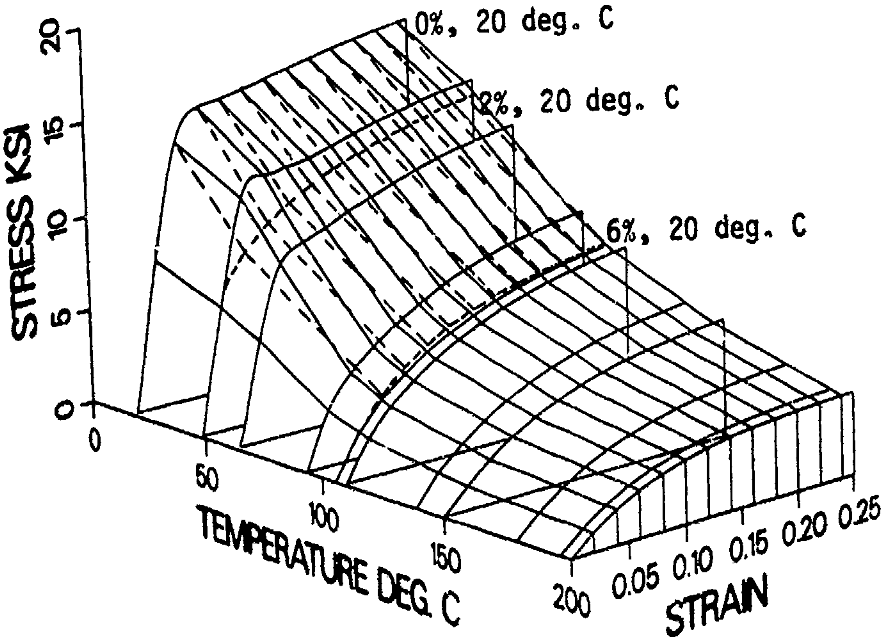
where a_{12} is the W-L-F time-temperature shift factor. Graphically, this quantity is the distance along the log t axis that a log E(+) vs. log t curve (or log D(t) vs. log t curve) at temperature "2" should be translated horizontially to coincide with the corresponding curve at temperature "1". The quantities f_2 and f_1 are the free volume to specific volume ratios at these two temperatures; "B" is a material constant. (The a_{12} quantity can also be considered as the ratio of relaxation times for the two temperatures.) An equivalent expression for a_{12} can be derived from (1), where a temperature difference term appears multiplicatively in the numerator and additively in the denominator; this latter form of representing time-temperature superposition has been confirmed by numerous experimentalists.

Experiments on Nylon 6/6 fibers [5] and Nylon 6 films [6] also show that (at constant temperature) time-moisture superposition exists; however results do not quantitatively match predictions based upon the free volume concept of (1) above. For example, Howard and Williams [5] compared their Nylon 6/6 time- temperature and time-moisture superposition data sets to reveal an empirical temperatue-moisture equivalence of 15 deg C per 1% moisture, but could predict this relation if total specific volume rather than free volume was used as the correlating parameter. For Nylon 6 films, Onogi, et.al. [6] suggest that the effect of free volume increase only dominates at higher moisture levels (say, above 1% moisture). Alternatively, instead of the free volume approach we are planning dynamic mechanical loss tests to directly quantify the dependence of material viscosity upon temperature and moisture, hence providing an independent prediction for the equivalencing of temperature and moisture effects on the mechanical behavior of Nylon 6/6.

FIGURE 11:

TEMPERATURE - MOISTURE EQUIVALENCING IN DIRECT COMPARISON (10^{-1}/sec)

NYLON 6/6, 0% MOISTURE
(11a)



NYLON 6/6, 0% MOISTURE
(11b)

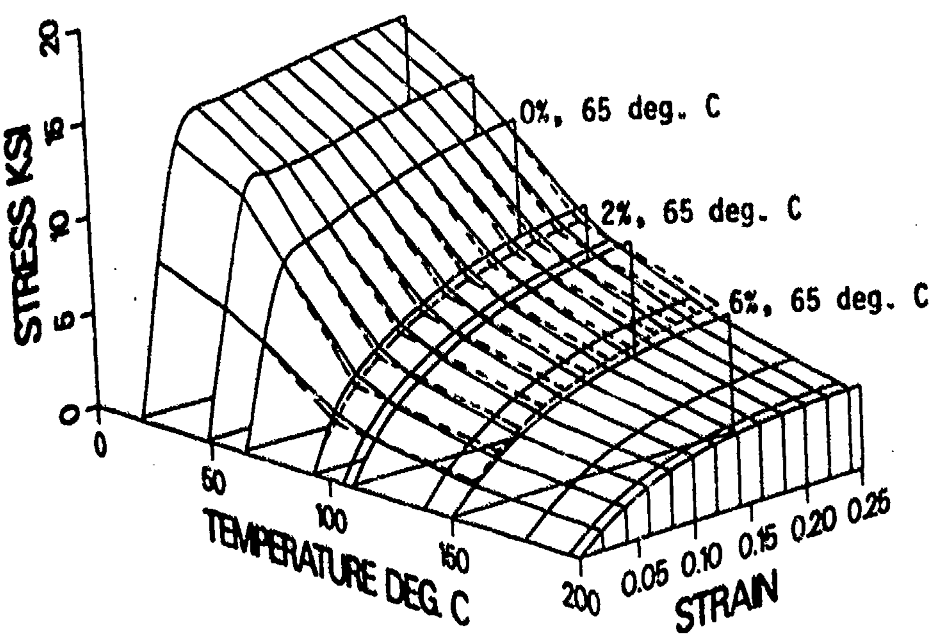
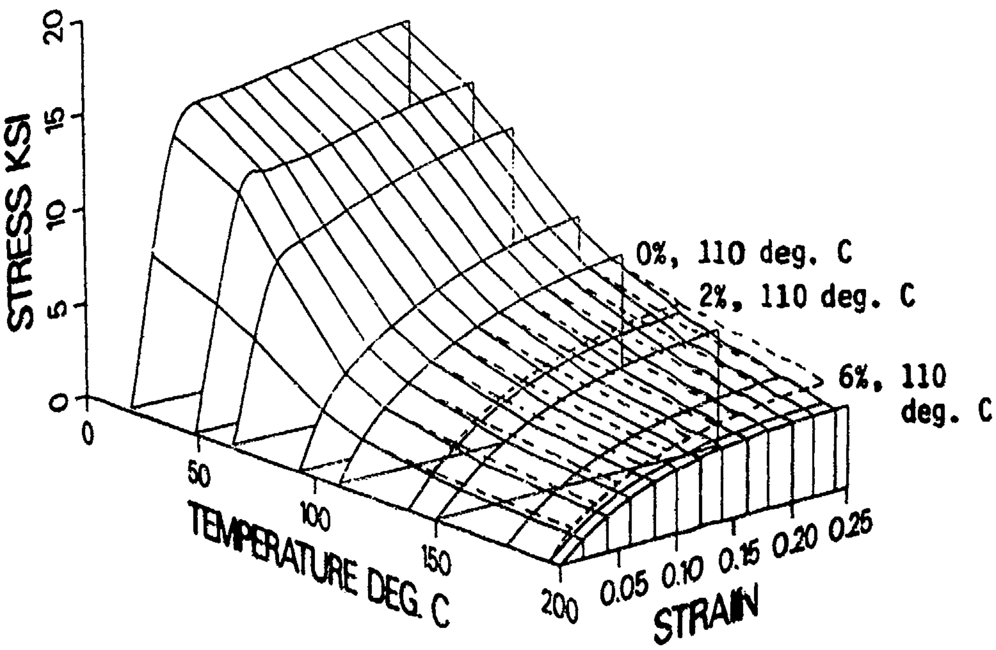


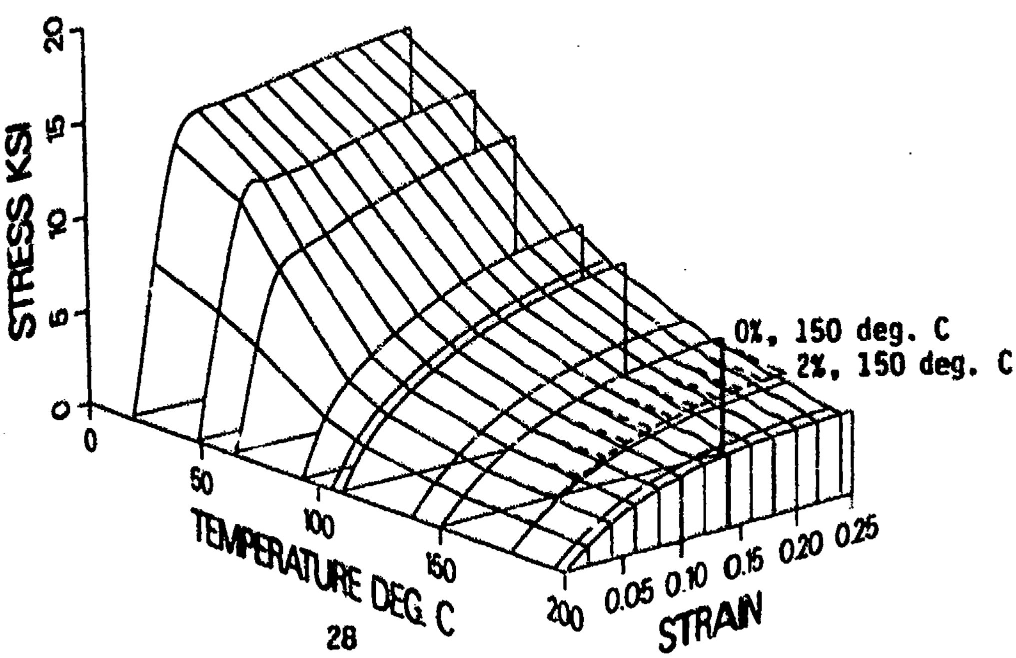
FIGURE 11 (Cont'd.)

TEMPERATURE - MOISTURE EQUIVALENCING
IN DIRECT COMPRESSION (10^{-1}/sec)

NYLON 6/6, 0% MOISTURE
(11c)



NYLON 6/6, 0% MOISTURE
(11d)



CONCLUSIONS

Material test results are presented for the nonlinear mechanical behavior of Nylon 6/6 in compression. Three deformation modes (direct compression, stress relaxation and creep) are considered and the effects of temperature, moisture and strain rate are investigated. The results indicate:

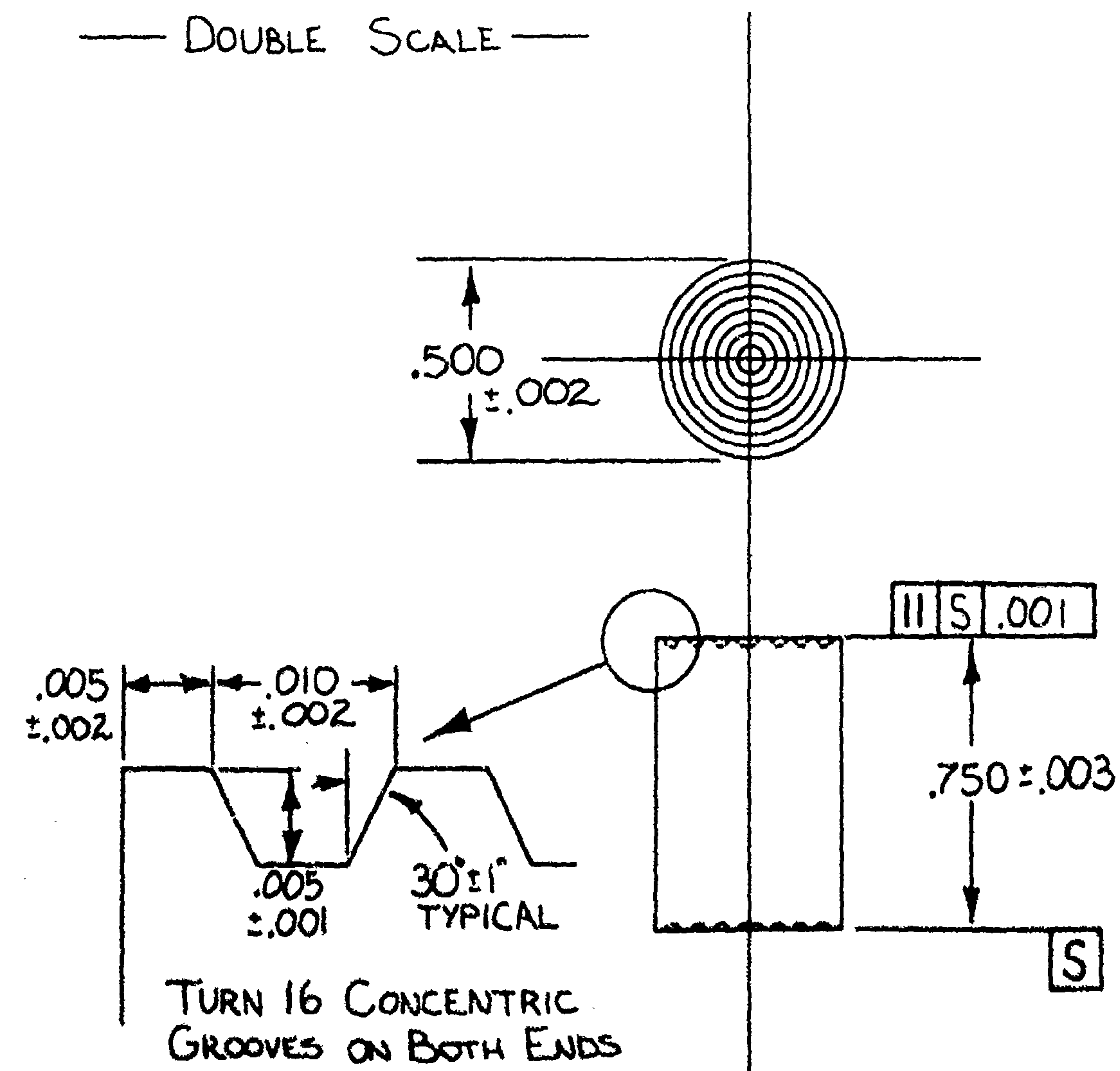
a. Elevated temperature lowers the overall strength, elastic modulus and strain-rate sensitivity; high moisture content lowers the strength and elastic modulus.

b. While elevated temperature and moisture both lower the elastic modulus, the influence of moisture is less pronounced at higher temperatures.

c. In direct compression, the similar effect of temperature and moisture is approximated by an equivalencing factor of 14 deg C per 1% moisture. This value carries over reasonably well to stress relaxation and creep modes.

APPENDIX

GROOVED COMPRESSION SPECIMEN



NOTE: REFACE IF NECESSARY AFTER GROOVING TO MAINTAIN PARALLELISM

DU PONT COMPANY
POLYMER PRODUCTS DEPARTMENT

CUSTOMER SPECIFICATIONS
PRODUCT CERTIFICATION TESTS

PRODUCT: Zytel® 101 NC-10 SPECIFICATION: MIL-M-20693B TYPE I

Tests	Procedure	Limits	Results
Batch Acceptance Inspection		LOT NO:	53 FR 01
Melt Point	ASTM D-789	250-260°C	259*
Specific Gravity	ASTM D-792 or D-1505	1.13-1.15	1.14*
Relative Viscosity	ASTM D-789	49 Min.	52.2
Moisture Content	ASTM D-789	0.28% Max.	0.19
Periodic Batch-Check Inspection		LAST TEST DATE:	August, 1985
Deformation Under Load @ 2000 psi	ASTM D-621	1.4% Max.	1.0
Stiffness	ASTM D-747	200,000 psi Min.	306,144
Tensile Strength	ASTM D-638	11,000 psi Min.	12,450
Elongation	ASTM D-638	50% Min.	63
Izod Impact Strength Notched	ASTM D-256	0.80 ft.lb./in. Min.	1.3
Heat Distortion Temp.	ASTM D-648		
@ 66 psi		182°C Min.	226
@ 264 psi		66°C Min.	90
Water Absorption	ASTM D-570	1.5% Max.	1.3

*Tested Annually

:is
DOC3221A

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